

THE ANALYST.

FEBRUARY, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Annual General Meeting of the Society was held at Burlington House on Wednesday, January 16. In the unavoidable absence of the President, Mr. Otto Hehner occupied the chair.

The minutes of the previous meeting were read and confirmed.

Mr. Herbert Stephen Shorthouse, 47, Pershore Road, Birmingham, was elected a member of the Society.

The accounts for the year, which had been audited by Mr. Kitto and Dr. Teed, were presented by the Treasurer, Mr. E. W. Voelcker. These showed that the Society was in a more satisfactory financial condition than in any previous year.

Referring to that portion of the accounts connected with THE ANALYST, the Sub-Editor, Dr. W. J. Sykes, said they had again had a successful year. Since the journal had been in the Society's hands, the receipts in connection with it had risen steadily and continuously, whilst the working expenses had diminished.

On the proposition of Dr. Stevenson, the report and accounts as presented were unanimously adopted.

On the motion of the chairman, a vote of thanks was passed to the President and Council of the Chemical Society for having allowed the Society the use of their rooms during the past year.

The valedictory address of the retiring President, Sir Charles A. Cameron, M.D. (whose duties as Medical Officer of Health detained him in Dublin), was then read by Dr. Bernard Dyer, Hon. Secretary.

RETIRING ADDRESS OF THE PRESIDENT, SIR CHARLES A. CAMERON, M.D.

GENTLEMEN :—Those who contend that the offices of Public Analyst and Medical Officer of Health should not be combined in the same person, may fairly claim to have, in my absence from the annual meeting of the Public Analysts' Society, a strong proof of the soundness of their views. In my capacity as President of the Society of Public Analysts, it is clearly my bounden duty to be present at their annual gathering. On the other hand, as the Medical Officer of Health for Dublin, it is imperative that I should be at my post whenever an epidemic is present. Unfortunately this is the case just now, for small-pox, absent for fourteen years from Dublin, is now spreading rapidly throughout the city. Whilst no member of our Society will suffer in health by the absence of the President from the annual meeting, the presence of the Dublin

health officer at his post at this juncture might possibly be the means of preventing some people from contracting one of the most loathsome of diseases. May I venture, therefore, to hope that my absence from your annual meeting will not be regarded as a proof of want of interest on my part in the affairs of our Society, but that it is due to unavoidable circumstances? I feel indeed most grateful to you for having allowed me to occupy for the period of two years the distinguished position of your President. Your kindness in this respect I regard, not merely as a compliment to myself, but also as one to the section of our Society who reside in Ireland. Realizing how little I have done to merit being placed in this position, I feel all the more sensible of the kindness shown to me by one and all of our members.

I feel under a great obligation to the ex-President, who has so kindly acted as my substitute during the last two years—I refer to my friend, Mr. Otto Hehner. I may without flattery say of him that the substitute was much superior to the person whose place he occupied in his knowledge of the affairs and objects of our Society.

I am rejoiced to think that I transfer the Presidency of our organization to a distinguished chemist of whom we all feel proud. He enters upon his duties at a time when our Society has reached the highest degree of prosperity to which we have yet attained. We are more numerous than we have ever before been. Our members are to be found throughout all the English-speaking countries. Our work compares favourably with that of a similar kind performed in any other country. The interest felt in our meetings during the year seems to have been fully maintained, and the papers read at them, together with the discussions which followed, have not fallen below the standard quality.

THE ANALYST, under its new management, exhibits a decided improvement, which is all the more remarkable, seeing that the cost of producing it has been decreased.

Our members have no doubt been much interested in the proceedings of the Committee of the House of Commons, who were appointed last session to inquire into the workings of the Sale of Food and Drugs Act. Up to the present the evidence submitted to the Committee has not on the whole been in favour of enacting a more stringent measure to abate adulteration. The Public Analysts have, however, yet to be heard, and we must hope their evidence will convince the Committee that to relax the provisions of the present rather weak Acts would not be to the advantage of the public at large, though it might benefit a few individuals. I trust that the outcome of the Committee's deliberations will be the adoption of a Bill substantially the same as that drawn up recently by our Society. I think that every effort should be made to induce the Committee to recommend the fixing of standards for all articles that admit of it. In prosecutions for excess of water in butter, the defendant's counsel or solicitor constantly raises the point that there is no legal standard in the case, and that consequently the Public Analyst has no right to fix one. The immense experience which the Public Analysts have in reference to the composition of food, their general scientific knowledge, and, I may add, their common-sense, are sufficient to enable them to determine what ought to be the maximal and minimal proportion of any of the variable constituents of food. We are not indebted to the dairy farmers for our knowledge of the minimal amount of non-fatty solids in milk. We cannot rely upon

them to tell us what is the maximal quantity of water which might fairly and properly be permitted in butter. For my part, I shall continue to hold that no butter, fresh or salted, need contain more than 16 per cent. of water. If through carelessness or want of skill larger quantities should be found, then it is the maker, and not the consumer, who should suffer. The following statistics in reference to the amount of water in Irish butter may prove interesting:

In my laboratory in 1893-94, 1,434 specimens of butter were examined, of which 503, or 35·1 per cent., contained more than 16 per cent. of water. In 1893, 549 specimens were examined. They came from the four Provinces, but those from Munster contained the largest proportion of water, as shown by the following table:

	Munster.	Leinster.	Ulster.	Connaught.
Number of samples	357	67	79	46
Percentage containing less than 16 per cent. of water	50·10	85·07	86·07	76·08
Percentage containing more than 16 per cent. of water	49·90	14·93	13·93	23·92

In 1894 the figures stood as follows:

	Munster.	Leinster.	Ulster.	Connaught.
Number of samples	604	179	50	52
Percentage containing less than 16 per cent. of water	57·61	86·60	92	82·70
Percentage containing more than 16 per cent. of water	42·39	13·40	8	17·30

Out of 961 specimens from Munster, 22 contained less than 9 per cent. of water; whilst in the 473 specimens from the rest of Ireland there were 43 containing less than 9 per cent. of water. No fewer than 87 of the 961 Munster specimens contained more than 20 per cent. of water, whilst in the 473 samples from the rest of Ireland, only 5 included more than 20 per cent. of water.

The largest percentage of water, namely, 34, was found in a Munster specimen.

In 1894 the percentage of water in the Irish butter that came into my laboratory exhibited a great improvement as regards excess of water as compared with 1893. This improvement may be due to the comparative coolness of 1893 as compared with the previous year. To some extent it perhaps may be accounted for by the great number of prosecutions for excess of water instituted in Ireland, chiefly in Munster. In the county Clare, for which I am Public Analyst, there were 70 successful prosecutions for excess of water in the half-year ended December 31, 1893. Within the last two years nearly 200 convictions have taken place on my certificate, nearly all in Munster. The magistrates who tried the cases were resident or stipendiary magistrates and justices of the peace. Many of the latter being country gentlemen, and farming more or less, were butter-makers themselves, yet with hardly an exception they adopted the statement in my certificate that 16 per cent. of water was the maximal amount that should be tolerated in butter.

A short time ago the decision of a Limerick Petty Sessions Court inflicting a fine for excess of water in butter was reversed by the County Court judge (Mr. Adams). This judge, who has a great reputation for wit, said that chemists were about the least reliable of witnesses!

Let us now return to the affairs of our Society.

Death was not very busy in our ranks. In 1894 our death-rate was only 0·8 per 100 of our members, or less than half the rate for the whole population. We have lost only two members, Dr. C. R. Alder Wright, F.R.S., and Dr. Emmerson. Dr. Wright, although not a Public Analyst, was a chemist and analyst of high reputation, and he took a great interest in the affairs of our Society. His loss to science will long be felt. Dr. Emmerson was one of our oldest members, and rendered good services in the districts (Leicestershire, Rutland, Northampton, Peterborough, etc.) for which he was Public Analyst.

In addition to our losses by death, we have had a few secessions and removals of members; but, on the other hand, our gains have exceeded our losses. In 1894 one honorary and ten ordinary members were elected, together with four associates. At present our Society is constituted as follows:

Honorary members	...	...	...	...	10
Members	...	...	...	...	210
Associates	...	...	...	...	31
Total					251

No fewer than twenty-four papers were read at the monthly meetings, and whilst all were more or less interesting, several of them were very valuable contributions to analytical chemistry. The following is a list of the papers read in 1894:

"Milk and Milk Products." By H. Droop Richmond.

"Note on the Detection of Cotton-seed Oil in Lard." By E. J. Bevan.

"The Loss of Total Solids in Milk on Keeping." By E. J. Bevan.

Notes from the Laboratory of the Royal Agricultural Society of England: (i.) "The Occurrence of a Poisonous Leguminous Seed in Indian Peas. (ii.) "Analyses of Waters from Wells in close proximity to Churchyards." By J. Augustus Voelcker, Ph.D., B.Sc.

"Some Frequently Neglected Errors of Analysis." By H. Droop Richmond.

"On Beef Stearin in Lard." By Otto Hehner.

"Note on Lemon and Orange Peel." By E. G. Clayton.

"Note on the Analysis of Phosphor Tin." By Frank L. Teed, D.Sc.

"A Simple Apparatus for the Extraction for Analysis of Gases dissolved in Water." By Sidney Harvey.

"On the Recognition of Exhausted Ginger." By A. H. Allen and C. G. Moor.

"On the Change in the Composition of Butter by Long Keeping." By A. H. Allen and C. G. Moor.

"The Examination of Urine for Sugar." By Alfred H. Allen.

"The Composition of Cheese." By W. Chattaway, T. H. Pearmain and C. G. Moor, B.A.

- "Note on the Valenta Acetic Acid Test." By W. Chattaway, T. H. Pearmain, and C. G. Moor, B.A.
- "The Detection of Methylated Spirit in Tinctures, Spirits, and other Compounds." By A. Ashby, M.B., F.R.C.S.
- "Roasted Chicory." By E. G. Clayton.
- "The Need for Fuller Statistics of Adulteration." By C. H. Cribb, B.Sc.
- "Note on Woody Fibre Determination." By A. P. Aitken, D.Sc.
- "Note on the Analysis of Sodium Peroxide." By L. Archbutt.
- "Note on Gladding's Process for the Determination of Resin in Soap." By L. Archbutt.
- "Note on a Sample of Red Water." By W. F. Lowe.
- "On Rechnagel's Phenomenon (the Rise in the S. G. of New Milk)." By H. Droop Richmond.
- "The Composition of Milk, and the Conditions Affecting it, as shown by Dr. Bell's Analyses." By J. F. Liverseege.

The preparation of a suggested Bill for the amendment of the existing Acts in relation to adulteration accupied no inconsiderable proportion of the time of our members who undertook the task. It is to be hoped that the Bill, or something closely resembling it, may be adopted by the Committee of the House of Commons, to whom it will be submitted. I would venture to suggest one amendment to the existing Acts, namely, that the Public Analyst should not be required to receive the whole of a sample of which the vendor has refused a part. When the inspector purchases or procures a sample, he ought to divide it at once into two parts; if the vendor refuses to accept a portion, one part should be sent to the analyst, and the other, sealed up at once, should be retained by the inspector for production in court. There appears to be no object gained by the analyst, and not the inspector, dividing the sample, except that unnecessary trouble is imposed upon the analyst. Of course this observation does not apply to the cases referred to in clause 15 of the suggested Bill. With respect to the transmission of articles to the analyst, the Irish Court of Queen's Bench have decided that they may be sent by railway. Bulky boxes of samples of beer, milk, etc., cannot well be sent by post.

It would be desirable to provide that when a mixture of an article with one much cheaper is sold, some indication as to the relative proportion of each constituent of the mixture should be given to the purchaser. I once certified that a mixture of 95 parts of chicory and 5 of coffee was not of the nature, substance, and quality of the article demanded. The magistrate who tried the case held that so long as there was any coffee in the mixture, it could not be regarded as an adulterated or debased article.

It is satisfactory to find that the principal recommendations of our Society for the amendment of the anti-adulteration Acts were approved of by the Congress held last autumn in London under the auspices of the British Institute of Public Health.

In bringing this short address to a conclusion, I must thank most heartily my friend Dr. Dyer, one of our energetic and zealous secretaries, for having during the last two years kept me posted up on all the affairs of our Society. I earnestly hope

that he and all the members of our Society may enjoy full measures of happiness and prosperity in 1895, and in many a succeeding year.

Mr. Hehner proposed that a very hearty vote of thanks be accorded to the retiring President for his interesting and valuable address, and for his services during his Presidency.

Dr. Stevenson, in seconding Mr. Hehner's proposition, which was carried with acclamation, said that the absence of Sir Charles, although much to be regretted, was easily excused under the circumstances.

Dr. Rideal and Mr. Cribb having examined the ballot-papers, the Chairman announced that the following gentlemen had been elected officers and Council for the ensuing year :

President—Thomas Stevenson, M.D., F.R.C.P.

Vice-Presidents (who have filled the office of President)—M. A. Adams, F.R.C.S. ; A. H. Allen ; Sir Chas. Cameron, M.D., F.R.C.S. ; A. Dupré, Ph.D., F.R.S. ; Otto Hehner ; Alfred Hill, M.D. ; J. Muter, Ph.D., M.A., F.R.S.E. (Who have not filled the office of President)—A. Wynter Blyth, M.R.C.S. ; Walter J. Sykes, M.D. ; John A. Voelcker, M.A., B.Sc., Ph.D.

Treasurer—E. W. Voelcker.

Hon. Secretaries—Edward J. Bevan, Bernard Dyer, D.Sc.

Other Members of Council—Charles E. Cassal, W. Chattaway, C. H. Cribb, B.Sc., W. W. Fisher, M.A., B. Kitto, H. Droop Richmond, F. W. Stoddart.

The names of those Members of Council whose term of office has not yet expired, and who consequently do not retire this year, are James Baynes, Sidney Harvey, W. Morgan, Ph.D., W. Cobden Samuel, and W. F. Keating Stock.

Dr. Thomas Stevenson, the newly-elected President, then took the chair amidst loud applause, and said that he desired to return his hearty thanks for the honour done him. He felt the position a somewhat difficult one, since he could not pretend to the qualifications which the late President possessed. Mr. Hehner had referred to the foundation of the Society in terms which brought many old friends back to his mind. It might not be known to all that he (Dr. Stevenson) was the first Treasurer of the Society. His mind went back twenty years, for he found that the first general meeting took place on February 5, 1875. He should endeavour always to be present at the meetings, though he could not promise never to fail, and to contribute as much as possible to the interests of the Society.

In the absence of the author, Dr. Dyer read the following paper :

NOTE ON THE USE OF MAIZE AS AN ADULTERANT OF OATMEAL.

By JOHN WHITE, F.I.C.

In the course of my work under the Sale of Food and Drugs Act, I have recently had submitted to me a large number of samples of fine oatmeal. Upon making a microscopical examination of these in the ordinary way, I noticed in several the presence of some granules larger than those of the oat, which, upon further investi-

gation, proved to be maize. This is to me an entirely novel adulterant of oatmeal, and I cannot discover any definite record of its previous use. The fact, moreover, that it does not appear to have been certified as an adulteration by any Analyst—at any rate recently—is my reason for bringing it before the Society.

There is of course no new chemical process to be described in this connection, but as the detection and estimation of a faceted starch such as maize, in the presence of oatmeal, is much more difficult than the recognition of a non-faceted starch like barley, a short description of the method I adopted may be useful to others.

The whole of the sample should be thoroughly mixed in a large mortar. Then take a small quantity, from 0.25 to 0.5 of a gramme; rub down in a small ground-glass mortar with from 5 to 10 c.c. distilled water, and while the particles are in suspension, take out a drop on the blade of a penknife and place on the slide. The estimation may now be made by comparing with standard mixtures put up in a similar manner. The micrometer may, of course, be used with advantage; but the most valuable information as to the amount of maize present is obtained by examining under polarized light, the black cross being shown perhaps better by this starch than by any other. I have found that an occasional oat granule will polarize, but the cross is naturally very much smaller than the one shown by maize and not so well defined, and very little practice is necessary to enable one to distinguish between the two with certainty. I obtained this result with oats ground by myself, and which had been supplied to me by the grower.

The amount of ash does not seem to give any clue to the presence of maize. In some of the adulterated samples it was rather higher than the average, but not abnormally so.

In considering the likelihood of maize becoming a regular adulterant of oatmeal, it is necessary to compare their respective market values. At the present time the two cereals are about equal in price in the raw state, and the vendor of one of these samples raised this point in his defence at the hearing. It must, however, be borne in mind that the proportion of meal to husk in maize is very much higher than in the oat, and consequently, equal weights of raw material being taken, much more maize-meal than oatmeal will be produced.

In neither of the samples was the amount of maize present large, the maximum I have yet found being 3 per cent. In some of the samples there were traces of barley starch in addition.

DISCUSSION.

Dr. Dyer said it was singular that the proportion of maize should be so small as 3 per cent. Of course, in a sample of home-grown oats the presence of maize could scarcely be accidental, but with foreign oats it was by no means remarkable. A great deal of grain of all kinds, including oats, was imported from maize-growing countries, and very often the quantity of total admixture or foreign matter was very considerable. Oats, for instance, were deemed commercially pure if the total amount of admixture was within 3 per cent. The foreign matter, which often amounted to very much more, consisted not merely of earth and chaff, but of other grains, including barley,

wheat, rye, etc., and occasionally a few grains of maize, so that small quantities of foreign starches might very well be found in the meal. He did not know if oats of this class were used for grinding; they were no doubt chiefly used for horses; but the facts he pointed to should lead analysts to be cautious.

Mr. Cassal said he had recently examined some official samples of oatmeal adulterated with barley. He had certified two samples to contain 10 and 15 per cent. of barley respectively. The adulteration of oatmeal with barley-meal was no doubt remunerative, but 3 per cent. was a curiously low percentage in a matter of this particular kind.

Dr. Dyer remarked that barley was a very cheap grain indeed. Foreign barley was somewhat dearer now, but during the last year could have been bought at 11s. a quarter. He, too, had examined samples of oatmeal adulterated with barley, in which the percentage of barley was considerably in excess of that reported by Mr. Cassal.

Mr. Cassal said that Mr. Hehner, who examined some of his samples, arrived at practically the same conclusion as himself. His authority did not prosecute in these cases.

Dr. Dyer said that the prices of grain fluctuated a great deal, and were different for home produce and for foreign imported grain. The price of malting-barley was high, but grinding-barley was cheap—only about one-half or one-third of the price of malting-barley, and much cheaper than good Scotch oats.

The two following papers were read by their respective authors :

NOTE ON A SAMPLE OF SUPPOSED LARD.

BY BENEDICT KITTO, F.I.C.

LARD, being a substance which easily lends itself to various forms of sophistication, has from time to time given public analysts a considerable amount of work; and it is no doubt advisable that any peculiarity observed in samples should be brought to the notice of the members of our society. It was suggested to me by our former president (Mr. Hehner) that a recent experience of mine should be so notified.

A sample of "lard" was submitted to me for analysis, under the Sale of Food and Drugs Act, which, when subjected to the iodine absorption test, was found to take up 14.96 per cent. only of iodine. Having thus found that it was not pure lard, Reichert's test was applied, and the $2\frac{1}{2}$ grammes operated upon required 4.4 c.c. of decinormal alkali. These results at once pointed to cocoa-nut oil. The sp. gr. at 100° C. was .868, and the melting-point 27.5°. The potassic hydrate neutralized was 22.95 per cent., showing a saponification equivalent of 244.

Having no cocoa-nut oil by me for comparative tests, I went round to Mr. Hehner's laboratory, and he very kindly supplied me with a deodorized sample, and, I may add, ever willing to place his great experience at the service of his brother analysts, offered to go through the iodine absorption and Reichert's tests on a

portion of the sample, at the same time pointing out to me the harshness observable upon pressing the knife against the cocoa-nut oil, as if it had a crystalline structure. This feeling was also very marked when I tried my own sample. Mr. Hehner's tests confirmed my own. There was a taste noticeable in the sample which I could not recognise, but no distinct smell.

Although most of the tests corresponded fairly well with the results obtained from the cocoa-nut oil, the iodine absorption was much higher, owing probably to the admixture of a small proportion of cotton-seed oil—Becchi's silver nitrate test having pointed, though not conclusively, in that direction—or possibly to a little lard oil.

I reported the sample to be almost entirely composed of prepared cocoa-nut oil, and therefore not lard at all; and I believe this was not disputed in court. A conviction of the seller followed.

So far as I know, this is the first case for some years where cocoa-nut oil has been detected doing service in this direction. About six years ago, Mr. A. H. Allen communicated to this society a case in which he found 33 per cent. of cocoa-nut oil as an adulteration in lard (ANALYST, xij., 189); but I have not heard of its having been noticed since that time until this sample was analysed.

NOTE ON A SAMPLE OF LARDERINE.

BY R. BODMER.

IN November last I received from one of my inspectors a sample labelled "Lardarine." The article had been exposed for sale as lard, but before completion of the purchase by the inspector the vendor declared it to be "lardarine"—a word coined in imitation of margarine.

The sample contained a trifling proportion of water. On analysis the following figures were obtained :

Melting-point	...	...	...	38° C.
Sp. gr. at 100° F. (water at 100° F. = 1)	...	...	...	0.9105.
Reichert (2.5 grammes)	...	...	...	1.0 c.c. $\frac{N}{10}$ Baryta.
Temperature reaction (Maumené)	...	...	...	43° C.
Iodine absorption	...	...	...	65.6 per cent.
Saponification 1 gr. required	...	...	...	201.8 mg. KHO.
Saponification equiv.	...	...	...	277.7
Nitrate of silver	...	...	...	copious reduction.
Crystallization from ether	...	...	...	beef crystals.
Ether deposit (Stock's process)	...	...	...	37 mg.
Oleo-refractometer	...	...	...	+ 6°*

The sample was evidently a mixture of beef stearin, cocoa-nut fat, cotton-seed oil, and *probably* some lard.

Taking the Reichert figure as indicating about 25 per cent. cocoa-nut fat, the presence of which is also confirmed by the saponification figure, and the E. W. D.

* Kindly taken for me by Mr. Bevan.

showing about 10 per cent. of beef stearin, and working out the iodine absorption, the following approximate proportions are obtained :

Beef stearin	...	...	10 per cent.	} Giving a calculated iodine absorption of 65 per cent.
Cocoa-nut fat	...	...	25 "	
Cotton-seed oil	...	...	45 "	
Lard	...	...	20 "	

Thus :

10 per cent.	beef-stearin	=	2.5	iodine.
25	"	cocoa-nut fat	=	2.25 "
45	"	cotton-seed oil	=	48.15 "
20	"	lard	=	12.00 "
64.90				

I understand that lardine is now a recognised article of commerce, and the question naturally suggests itself whether the Margarine Act should not be extended so as to make it a penal offence to expose for sale a spurious lard without the same being labelled "Lardine," or some such term.

DISCUSSION.

Mr. Richmond asked Mr. Bodmer to say what strength of acid was used in his Maumené test, and how it was done, so that they might compare it in their own minds with their own ways of doing it. He would also like to have some figures obtained by the same strength of acid for one or two well-known oils, such as olive oil and cotton-seed oil.

Mr. Cassal wished to know what Mr. Bodmer meant by "beef crystals." There might be different opinions as to the characters of such crystals, and some views as to those characters which had been prematurely accepted were erroneous. It was useless, for instance, to examine the crystals obtained by a third crystallization.

Mr. Hehner said that lard substitutes had been sold for many years without any secrecy, to which, if they were sold under their proper titles, there could not be any reasonable objection.

This sample was a peculiar one ; its iodine absorption was lower than is usual with lard substitutes, and it had apparently been made up with the object of giving a normal iodine absorption. He doubted whether Mr. Bodmer's sample really contained lard, because it seemed somewhat unreasonable to put much lard into a mixture sold as a lard substitute.

The other sample, which he had had the privilege of seeing, was very much like pure cocoa-nut oil with a very small admixture of other substances. Purified cocoa-nut oil was well known ; but he had never before heard of its being used or sold almost pure for lard. It seemed, however, to be a promising lard substitute, and, personally, he should prefer a pure vegetable substance to a mixture of doubtful composition.

Mr. Bodmer said he performed the Maumené test with half the quantities recommended by Mr. Allen, because the samples he received were usually small. As a rule the results were comparable with those obtained by Mr. Allen. The rise in temperature was 40° C. for olive oil, and about 75° C. for cotton-seed oil.

His statement as to beef stearin must be taken with a certain amount of faith. The crystals he obtained were distinctly plumose, like those obtained from actual beef stearin. The amount of ether-washed deposit corresponded, according to Stock's table, with about 10 per cent. of beef stearin. From the melting-point and the iodine number it was pretty certain that beef stearin was present.

Mr. Hehner remarked that beef stearin would undoubtedly be present; in fact, it was a necessary constituent of such mixtures.

Mr. Cassal said he was not expressing doubt as to the presence of beef stearin in the sample. He wanted to know upon what evidence the statement was founded. Plumosity alone was not sufficient evidence to prove the crystals to be beef crystals.

After the meeting the annual dinner took place at the Criterion, Piccadilly.

THE DETERMINATION OF WOODY FIBRE IN FODDERS.

BY A. P. AITKEN, D.Sc.

(Read at the Meeting, Dec. 5, 1894.)

THE name woody fibre is a conventional one, including under it a considerable number of substances, which agree in this—that they are composed of carbon, hydrogen and oxygen, and are insoluble in dilute solutions of acid and alkali when boiled in these for a certain time.

The generally recognised method of estimating woody fibre is to boil the finely-ground substance in a solution of $1\frac{1}{4}$ per cent. sulphuric acid for half an hour, maintaining the solution at the same strength all the time. The substance is then washed till quite free from acid, and thereafter boiled for half an hour in a $1\frac{1}{4}$ per cent. solution of caustic soda maintained at a constant strength, and after thorough washing the residue is dried at about 110° C. and weighed. This substance contains a certain amount of ash, and usually a small amount of albuminoid matter, both of which are determined and their weight deducted. The residue is regarded as woody fibre. This is, of course, a purely conventional method, laying no claim to scientific accuracy, nevertheless yielding information which is of some value in determining the purity or quality of a fodder, and is of some use as a rough guide in determining its feeding value.

My object in the present note is not to discuss the value of the method itself, or the information which may be obtained by means of it, but simply to direct attention to a modification in the practical carrying out of the process that will be found to make it simpler and less irksome, and to yield results that are more uniform than those usually attained. There is no item in the analysis of a fodder wherein analysts differ so widely as in the woody fibre. It is no uncommon thing to find in two analyses of the same fodder by different analysts that the one has found double the amount of woody fibre that the other has when the total amount is comparatively small.

This may arise from their using different strengths of acid and alkali, or from their continuing the boiling for a longer or shorter time. Since the quantity of woody

fibre found varies according as the solutions are stronger or the times of boiling are longer, this is one of those matters upon which analysts should come to an agreement and adopt a prescribed method of working, so that the results obtained may at least be comparable, and the simpler the method prescribed, the more likely is it to be adopted.

Some analysts use 5 per cent. solution of acid and alkali, and some prefer $2\frac{1}{2}$ per cent. solutions; but $1\frac{1}{4}$ per cent. solutions are more convenient, as they are less liable to frothing and more easily washed out of the residue. The results obtained by using stronger solutions are in most cases less than those obtained by using the weaker ones, but the weakness of the solutions used can be compensated by increasing the duration of boiling. The maintenance of the strength of the solutions during boiling is achieved either by adding water from time to time as they evaporate, or by attaching the flask in which the process is carried out to a return-flow condenser. When a large number of estimations are going on at once, neither of these methods is convenient, and when flasks are used in which to conduct the operation, the frothing of the contents causes much trouble. This is got over, or at least lessened, by introducing a blast of air into the flasks, but that also is attended with inconvenience. I get over the difficulty entirely by abandoning the use of flasks altogether, and by abandoning the boiling also. I now use beakers, which I immerse in a steam-bath, and which I cover with watch-glasses. The result is that the solutions are maintained at the temperature of boiling water and no evaporation takes place, so that the strength of the solutions does not alter, and the subsequent washing of the residues and their final removal from the beaker to a filter or Gooch crucible is easy. The extraction of the soluble matters is not as rapid when the solutions are simply subjected to the temperature of the steam arising from boiling water, but the difference is not very great—three-quarters of an hour in the steam-bath is equivalent to half an hour's boiling. During the operation the solutions require no attention, there is no frothing, no evaporation, no breaking of flasks, as is not unfrequent with the boiling process. The bath I use is simply a water-bath of elongated shape, at the bottom of which is a gutter kept automatically full of water, and only about an inch deep. The water is kept constantly boiling by means of little jets of gas issuing from a number of small holes bored in the top of a horizontal piece of brass tubing extending the whole length of the gutter, and fixed below it at a distance of about an inch and a half. The gas-jets are about two inches apart, and less than an inch high. The lid of the bath is removable, and is perforated with holes, into which the beakers are let down and suspended by their rims, and the bottoms of the beakers do not quite reach the level of the water-gutter. The whole body of the beaker is thus immersed in steam at about 100° C., and the contents of the flasks are subjected to perfectly uniform conditions, so that successive determinations of woody fibre so conducted give results of great uniformity, and are attended with less trouble than the boiling methods commonly practised.

In the case of oil-cakes, it is advisable to extract the oil with ether before setting about the determination of the woody fibre. A sufficiently complete removal of the oil can be effected in a very short time, as the small residue will be saponified during the alkali treatment.

Unless the bulk of the oil is removed before treatment, the fibre left after treatment with acid and alkali will contain a considerable residuum of oil; if, therefore, the oil contained in the sample operated upon has not been extracted before the fibre determination, the fibre obtained must have the oil extracted from it before weighing.

DISCUSSION.

Mr. Allen remarked upon the laxity which existed in speaking of the strengths of acids and alkalies. Percentages by volume and percentages by weight were often confounded, which led, of course, to grave errors and misunderstandings. Thus, if sulphuric acid of $2\frac{1}{2}$ per cent. by weight were prescribed, and the operator used water containing $2\frac{1}{2}$ per cent. by measure, he would be employing an agent containing about $4\frac{1}{2}$ per cent. by weight of the acid, and hence of nearly twice the prescribed strength.

Mr. Sidney Harvey agreed with Mr. Allen as to the importance of correctly and clearly defining the strength of solutions. He had for many years adopted the plan suggested by Dr. Aitken for heating, and his results were generally very fairly comparable.

Dr. Dyer said that in his own laboratory the custom was to boil up the material in a beaker over a low flame, from time to time adding a little water from a wash-bottle when the liquid boiled down, a mark on the side of the beaker serving as a guide. Although a crude way of work, it answered very well if it was watched, but attention was necessary to prevent any adherence of fibre to the sides of the beaker, which was apt to cause something like caramelization, leading, if unnoticed, to discrepant results. He thought, however, that errors were more usually caused by incomplete extraction of the oil from the fibre, either before or after treatment with acid and alkali. Mere alkaline treatment failed to saponify more than a fraction—sometimes only a moderate fraction—of the oil.

The Preparation of Litmus Solution. W. Schafer. (*Apoth. Zeit.*, 1894, ix., p. 839; through *Chem. Zeit.*)—Litmus solution of good quality can be prepared as follows: The dye stuff in cubes, as commonly sold, is placed in a percolator and extracted with distilled water. The extract is evaporated until it equals the weight of the original litmus, then treated with three times its weight of 90 per cent. alcohol, made strongly acid with hydrochloric acid and allowed to stand for two days. Azolitmin is precipitated in brown clots, while the accompanying dirty-violet colouring matter remains dissolved in the alcohol. The precipitate is collected on a filter and washed two or three times with acidulated hot water, until the filtrate, which is faintly red in colour, becomes on treatment with ammonia pure blue, without any violet tinge. The purified azolitmin thus obtained is brought into solution in water made faintly alkaline with ammonia, and the solution is diluted with distilled water until it is equal to three and a half times the weight of the original litmus. It is then accurately neutralized and preserved by the addition

of 10 per cent. of alcohol. The litmus solution thus prepared leaves nothing to be desired, its change of colour, when used as an indicator, being perfectly sharp.

B. B.

The Concentration and Distillation of Hydrogen Peroxide. R. Wolffenstein. (*Ber.*, 1894, xxvii., 3307; through *Chem. Zeit.*)—According to the experiments of the author, hydrogen peroxide is much more stable at moderately high temperatures than is generally supposed. The instability of this substance, which is often observed, must be referred to the presence of certain definite impurities. In order to secure its stability, hydrogen peroxide must be free from any substance capable of giving an alkaline reaction, from all traces of compounds of the heavy metals, and from solid substances of all kinds, even those usually accounted chemically indifferent. Distillation experiments conducted with hydrogen peroxide, complying with these requirements, show that the vapour is stable at moderate temperatures. Moreover, a 3 per cent. solution of hydrogen peroxide may be concentrated to about 45 per cent. without any considerable quantity volatilizing. When, however, this concentration is reached, the rate of volatilization rapidly increases, until at a pressure of 68 mm. and a temperature of 85° C., a product which is perfectly pure and almost anhydrous passes over. Hydrogen peroxide thus obtained is a clear, syrupy liquid, evaporating when exposed to air, and giving rise to a stinging sensation and the formation of white spots when brought in contact with the skin. It is *acid* to litmus, although dilute solutions are neutral to that indicator. The simplest method of preparing pure hydrogen peroxide from the 3 per cent. solution commonly sold consists simply in distilling the entire quantity. When larger quantities of more concentrated solution are required, the commercial peroxide (3 per cent. strength) is concentrated on the water bath to about 20 per cent.—care being taken that the preparation is free from the impurities mentioned above—and continuing the concentration in vacuo up to about 50 per cent., extracting with ether, and finally distilling in vacuo. By fractional distillation, hydrogen peroxide of any desired concentration may be thus obtained.

B. B.

Fallacies of Post-Mortem Tests for Morphine. Daniel L. Davoll, jun. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 12, pp. 799-808.)—The experiments here detailed were made to determine to what extent cadaveric alkaloids might respond to the usual tests for morphine. For this purpose the internal organs of a dog, which had been buried in a tight pine-box for forty-five days, were extracted by a modification of the older method, and the extract tested with the ordinary morphine reagents.

Bromine water, Wagner's solution, Mayer's reagent, and sodium phosphomolybdate all gave strong precipitates, and a cloudiness was obtained on the cautious addition of ammonium hydrate. The most satisfactory tests were Fröhde's reagent, Le Fort's test, and ferric chloride, all of which gave a negative result with the pure extract, while they would detect as little as 0.00005 gramme of added morphine. Of the three, Fröhde's reagent was the most sensitive. Iodic acid gave

indications of a reducing agent in the extracted matter, and HNO_3 and ferric chloride with potassium ferricyanide were quite useless.

During the process of extraction of the different organs a decided odour, resembling that of tri-methylamine, was given off.

C. A. M.

On the Gunning Method for Total Nitrogen in Fertilizers. W. E. Garrigues. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 12, pp. 795-799.)—In the estimation of fertilizers containing nitrates the Gunning method, as ordinarily applied, gives too low results. This is clearly shown in the following determinations, for which a mixture of pure sodium nitrate and tankage was prepared. The latter was valued by the ordinary Kjeldahl and Gunning methods and by soda-lime combustion with the following results. Percentage of nitrogen :

Kjeldahl.	Gunning.	Soda-lime.
7.05	7.05	7.08

The nitrogen in the mixture was then determined, and gave :

Theory.	Kjeldahl.	Gunning.
8.97	8.90	7.17
		7.31
		7.46

The author's explanation of the lower results obtained by the Gunning method is that the potassium sulphate, being added at too early a stage, protects the sample from the attack of the acid.

To avoid this, the following method of proceeding is recommended : The salicylic acid mixture is first added, and when the sample has been thoroughly wetted, the flask is allowed to stand for fifteen minutes, after which the thio-sulphate, is added in four or five portions with constant shaking. Each portion should be completely dissolved before the next is added. The potassium sulphate is then introduced, and the whole digested as usual.

When treated in this way the above-mentioned mixture gave :

8.85 8.87 8.90 8.92 per cent. of N.

Other mixtures gave equally satisfactory results, but in the case of sodium nitrate alone, neither by the Gunning nor the Kjeldahl method could a sufficiently accurate determination be obtained. A carefully prepared sample of the salt containing by theory 16.49 per cent. of nitrogen yielded :

Kjeldahl.	Gunning.
16.05	15.80
16.35	16.00
	16.15
	16.22
	16.38

No means of avoiding this loss could be found, and it would thus appear justifiable to take the highest figure in closely agreeing duplicates, instead of the usual average.

To avoid loss by frothing in the distillation, it is advisable to use a 32-ounce wash-bottle, instead of the 500 or 600 c.c. flask. C. A. M.

[EDITORIAL NOTE.—The Kjeldahl-Jodlbauer-Gunning method, skilfully worked, gives without difficulty *the whole* of the nitrogen in nitrate of potassium or sodium, or even nitrate of ammonium, whether alone or in admixture with other forms of nitrogen found in fertilizers. The sulphonated salicylic acid (or phenol) mixture must be poured *rapidly* on to the substance, after which reduction is performed with zinc dust gradually in the cold first, and then with but very gentle heat, till all zinc is dissolved. A drop of mercury is desirable, but not absolutely necessary. The whole is boiled for a time, after which, *and not till then*, the sulphate of potassium is added.—B. D.]

The Determination of Melting-Points and the Composition of some Candle Material. W. E. Garrigues. (Jour. Amer. Chem. Soc., 1894, xvi., 12, pp. 825-832.)

—For the purpose of obtaining concordant results by different observers, a modification of Dalican's method of taking the solidification point is recommended. 30 to 50 c.c. of the dried sample are placed in a small beaker, the thermometer bulb inserted in the melted mass, and the glass and its contents turned in one direction until the mercury stops receding or ascending. With stearic acid there is a rise of about 0.3° , and the temperature remains constant for about a minute. With paraffin wax there is no rise, but the mercury remains constant about half a minute, and then recedes at the rate of about 0.1° to 0.2° per minute. With mixtures of the two there is great diversity, ranging from no rise at all to one of nearly 4° . The latter represents a mixture of $\frac{2}{3}$ stearic acid to $\frac{1}{3}$ wax. The following table gives the melting-point and behaviour of the mercury in the case of mixtures in different proportions:

Stearic Acid.	Paraffin.	Melting-Point. Degrees.	Mercury. Degrees.
100	0	54.6	0.1 rise
100	10	53.4	No rise
100	20	52.4	"
100	30	51.6	"
100	40	50.8	0.05 rise
100	50	50.1	0.2 "
100	60	49.2	0.6 "
100	70	48.2	0.8 "
100	80	47.4	2.8 "
100	90	45.6	0.6 "
100	100	45.2	0.8 "
90	100	45.8	No rise
80	100	46.3	"
70	100	46.9	"
60	100	47.3	"
50	100	47.9	"
40	100	48.6	"
30	100	48.9	"
20	100	49.6	"
10	100	50.4	"
0	100	50.8	"

In cases where no rise of the mercury takes place, the twirling should be continued until a homogeneous semi-solid consistency is attained. If still no rise occurs, the temperature at which there was a pause is noted.

The latter part of the paper is chiefly concerned with the changes which take place in the distillation of the fatty acids of the fats used in candle-making. The conclusion arrived at is that the stearic and palmitic acids distil without change, while the oleic acid partly splits up into liquid hydrocarbons, tar, and a saturated or non-iodine consuming fatty acid.

C. A. M.

On the Technical Analysis of Asphaltum. Laura A. Linton. (*Jour. Amer. Chem. Soc.*, 1894, xvi., 12, pp. 809-822.)—It is usual to designate the constituents of bitumens as "Petrolene" and "Asphaltene" according to their behaviour with different solvents. The determination of these is purely empirical, but for want of a more scientific method is employed in this paper. In general terms, *asphaltene* is that portion of different forms of bitumen which is soluble in CS_2 , chloroform, benzene, and a few other less well-known liquids, and is not soluble in the menstrua that dissolve petrolene. *Petrolene* includes a large number of hydrocarbons, and is much less definite in character than asphaltene. Ethyl ether and the so-called petroleum naphtha are the solvents generally used for it. Of twenty-three asphalts examined in their relation to the solvents, turpentine, CS_2 and chloroform, all yielded some asphaltene to chloroform after previous treatment with hot turpentine; and in several CS_2 following turpentine dissolved a considerable quantity. It was found that Californian naphtha and ethyl ether could not be used as solvents for petrolene, since they also dissolved some of the asphaltene, and petroleum ether was therefore used.

The following method of analysis is recommended: Two portions of the asphalt are weighed in Erlenmeyer flasks; 50 c.c. of petroleum ether are added, and next morning decanted on a counterpoised filter placed in a three-inch funnel with a stopcock in its neck. The residue in the flask is digested with successive portions of petroleum ether until the liquid decanted through the filter ceases to be coloured. The whole of the bitumen is then transferred to the filter, washed well with petroleum ether and dried, together with the counterpoised filter, in a steam bath. The loss in weight of the bitumen represents the petrolene. The flask should also be dried and weighed, and any increase in weight deducted from the amount determined as petrolene. To determine the asphaltene, the substance on the filter is digested firstly with successive portions of hot turpentine, and secondly with chloroform, until the filtrates are colourless. During the digestion, the stopcock in the neck of the funnel is kept closed. The flask should also be rinsed out with the same solvents, and the washings poured over the filter. The loss in weight after drying the latter represents the asphaltene.

The filter is then burned in a platinum crucible, and the loss in weight represents the organic matter not bitumen.

The method is capable of yielding closely concordant duplicate results at each step. A typical analysis gives :

Water.	Petrolene.	Asphaltene.	Other Organic Matter.	Mineral Matter.	Total.
2.029	32.4455	22.1115	8.1215	35.2865	99.994.

C. A. M.

On the Chemical Composition of Cola-nut. C. Uffelmann and A. Bömer. (*Zeit. für angewand. Chemie*, 1894, xxiii., pp. 710-712.)—The mean results of the analysis of ten samples of Cola-nut, varying in price from 4s. to 20s. per pound, were :

Water.	Total Nitrogen.	Protein.	Caffeine (including Theobromine).	Ethereal Extract.
13.35	1.53	5.91	2.08	1.35
Starch.	Tannin.	Cellulose.	Other Extracted Matter (non-nitrogenous).	Mineral Matter.
45.44	3.79	7.01	18.21	2.9

In spite of the great difference in price, the different specimens showed a remarkable similarity in composition.

Of the inferior plants used to adulterate Cola-nut, only two, according to the analysis of Heckel and Schlagdenhauffen, contain caffeine, viz., *Cola Ballay* nut, 1.05 per cent., and the nut of *Cola gabonensis*, 0.263 per cent. The other usual adulterants are quite free from the alkaloid.

C. A. M.

Contribution to Milk Analysis. Ernst Beckmann. (*Report of Thirteenth Assembly of the Bavarian Association of Chemists*, 1894, pp. 16-18.)

(a) *Estimation of Fat.*

The disadvantages of the usual methods of extraction may be avoided by the following process, the object of which is to remove only such a quantity of the water that the ether can readily dissolve all the fat, but not so much that an emulsion is formed. This is most readily effected by precipitation of the casein and fat by means of lead acetate, and separation of the greater part of the serum by filtration.

Twenty-five c.c. (or, in very accurate determinations, a weighed quantity) of the milk are diluted with an equal volume of water in a stoppered glass cylinder. To the mixture 2.5 c.c., or, in the case of sour milk, up to 5 c.c., of official lead acetate solution* are added, the cylinder well shaken, and, finally, sufficient NaHCO_3 solution introduced to cause the precipitate to coagulate. After filtration, the residue on the paper is placed in a glass cylinder, together with small glass balls of about 3 mm. diameter, and shaken out with ether. The object of the glass balls is to prevent an emulsion, and to facilitate the solution of the fat in the ether. The filter-paper is also shaken out with ether in a second cylinder, and the solution added to the other one. The dissolved fat is then estimated either by distilling off the ether or by separating it by dilution. The process is rapid, and is equally reliable with every description of milk.

* One part lead acetate, three parts litharge, and ten parts water.

The following table gives the percentage of fat found in different specimens of milk, with control results by the Soxhlet method:

			New Method.	Soxhlet.
(1)	Fresh milk	...	4.065	4.07
(2)	Fresh skim milk	...	0.91	0.85
(3)	Sour milk	...	2.93	2.99
(4)	„ „	...	2.92	2.88 and 3.02
(5)	„ „	...	3.65	3.65
(6)	Sour skim milk	...	1.07	0.970

(b) *Freezing-Point.*

Since the freezing-point of a fluid depends on the nature and quantity of the substances dissolved in it, and not on those of the bodies in suspension, the determination of the freezing-point of milk will furnish a check result influenced by the amount of water present, but not by that of the fat. The normal freezing-point of milk is very constant, ranging from -0.54° to -0.58° , the mean being -0.554° . The lowering of the freezing-point below that of water is in proportion to the concentration of the milk.

The determination is made by placing the milk in a suitable vessel, surrounded by a mixture of ice and salt, inserting a thermometer, and constantly stirring until the solidification point is noted. At first the temperature falls from one to two degrees below the freezing-point, but when the separation of ice commences, rises again, and remains constant for some time. This is taken as the point of solidification. By the use of a thermometer graduated in one-hundredths of a degree, the reading is simple, but since an addition of 10 per cent. of water to a milk only alters the freezing-point by five and a half hundredths of a degree, it is to be feared that such a difference is too small to draw a conclusion from when the results are not concordant. In the case of sour milks and milks to which salts have been added, the depression of the freezing-point is increased, but in such cases the simultaneously increased electrical conductivity will correct the conclusion.

The author hopes to produce a freezing apparatus in a handy form, to serve as a check in milk analysis.

C. A. M.

On a Method of Estimating Sulphocyanic Acid. M. T. Goudoin. (*Journ. de Pharm. et Chimie*, 1894, ii., pp. 481-484.)—The acid is estimated as cuprous sulphocyanide, care being taken that no free HNO_3 is present. The liquid, if alkaline, is made acid with SO_2 solution (density, 1.005), and a 10 per cent. solution of pure CuSO_4 added so long as a precipitate forms. It is then heated at 80° until the blue tint appears, when the precipitate is filtered on a tared filter, washed till no longer acid, dried at 100° , and weighed.

The author's experiments prove that the cuprous sulphocyanide is stable in the presence of the two reagents, CuSO_4 and SO_2 ; and that the method is applicable in the presence of all salts usually met with in industrial waste waters. In a test mixture containing CaCl_2 , NaCl , Na_2CO_3 , MgCl_2 and 0.10 per cent. of potassium sulphocyanide, a precipitate of cuprous sulphocyanide was obtained corresponding to (1) 0.1004 per cent. and (2) 0.0994 per cent. of the potassium salt.

Cyanides do not interfere with the result.

C. A. M.

Estimation of Gelatin and Albumin in Peptone. Ernst Beckmann. (*Report of Thirteenth Assembly of Bavarian Chemists*, 1894, pp. 18-20.)—A method for the estimation of gelatin in meat extracts is based upon the fact that formaldehyde combines with it to form a non-fusible and insoluble compound—formalin-gelatin. In order to render insoluble one gramme of gelatin dissolved in water, about two drops of the 40 per cent. solution in water of formaldehyde (formalin) are added. The quantity required is so trifling that its weight may be neglected.

The presence of much free acid hinders the reaction, which, however, takes place perfectly well in a slightly alkaline solution :

- (1) 0.235 gramme gelatin, dissolved in a little water, furnished 0.209 gramme formalin-gelatin.
- (2) 0.274 gramme gelatin, dissolved in a little water, furnished 0.231 gramme formalin-gelatin.

If formic or acetic acid be present, either too little precipitate is obtained, or none at all.

- (1) 0.1840 gramme gelatin in 10 c.c. H_2O , treated with ten drops of formic acid, and left in the water bath ten minutes, yielded 0.0866 gramme.
- (2) 0.1256 gramme gelatin, on adding ten drops acetic acid with the formalin, yielded 0.0496 gramme insoluble residue.
- (3) 0.1420 gramme with 1 c.c. acetic acid gave no insoluble residue with formalin.

Soluble egg-albumin and serum albumin left residues on mixing with formalin.

- (1) 0.3680 gramme soluble egg-albumin gave 0.3408 gramme residue.
- (2) 0.3040 gramme soluble serum albumin gave 0.2810 gramme residue.
- (3) A mixture of 0.308 gramme gelatin and 0.2524 gramme egg-albumin gave 0.5402 gramme insoluble residue.

Merck's dry peptone was found to be completely soluble in the presence of formalin, and by this means gelatin and albumin could be easily separated from peptone.

- (1) A solution containing 0.294 gramme gelatin and 0.49 gramme peptone gave 0.256 gramme residue.
- (2) A solution containing 0.272 gramme serum albumin and 0.557 gramme peptone gave 0.265 gramme residue.

In determining whether a meat-extract contains gelatin, the albumin is estimated in an aliquot part of a watery solution by means of acid. Another portion is treated with formalin, steamed on the water bath, and, after boiling for a short time with water, the residue is collected in a Gooch's crucible, dried at $100^{\circ} C.$, and weighed. After subtracting the amount of albumin previously found, this gives the gelatin. The peptone, etc., in the filtrate can be precipitated in the usual way. The method will be of use in milk analysis for detecting adulteration with an emulsion of gelatin and fat.

C. A. M.

Oils. G. de Negri and G. Fabris. (*Zeit. für Analyt. Chemie*, 1894, Heft 5, pp. 547-574. *From the Italian.*)—The contribution contains the quantitative figures and details of the colour reactions of olive-oil and forty different kinds of seed-oils.

Of the 213 samples of olive-oil examined, the authors obtained 160 from the fruits, the remainder being procured from reliable manufacturers. In pressed-out oils the melting-point of the fatty acids varied from 24° to 27° C. In extracted oils, which also contained more stearin, it lay between 25° and 29° C. The iodine number was between 79.5 and 88 per cent., and the saponification equivalent between 185 and 196. In no case did Becchi's test give any colour with pure olive-oil, which had been repeatedly filtered. The general conclusion arrived at is that no single test is sufficient to decide whether all olive-oil is pure. In certain specimens of olive-oil traces of arachidic acid are said to have been found.

In the case of earth-nut oil no colour reaction could be obtained by Becchi's, Milliau's, Baudouin's, or Schneider's tests, but the rapid solidification of the soaps in taking the saponification equivalent was not observed in any other oil.

In the application of Becchi's test for cotton-seed oil, the two solutions recommended by the Italian Commission were used :

(1) One gramme AgNO_3 in 200 c.c. of 98 per cent. ethyl alcohol, 40 c.c. sulphuric ether, and 0.1 gramme HNO_3 .

(2) Fifteen c.c. colza oil in 100 c.c. amyl alcohol.

Ten c.c. of the oil to be examined were mixed with (1) 1 c.c. AgNO_3 solution and (2) 10 c.c. of colza solution added. After being well stirred, the mixture was divided into two parts, one of which was heated for fifteen minutes in boiling water, the other being left cold.

With cotton-seed oleo-margarine no coloration was obtained by Becchi's or Milliau's reactions. The fatty acids melted at from 27° to 30° C.; the iodine number was 8.9 per cent.; and the saponification equivalent 257. The authors, therefore, conclude that its presence in butter and lard cannot always be detected. C. A. M.

On the Detection of Small Quantities of Metals of the Second Group in Precipitated Sulphur. R. Fresenius. (*Zeit. für Analyt. Chemie*, 1894, Heft 5, pp. 573-574.)—To the fluid containing the separated sulphur a layer of about 2 cm. of benzene or petroleum ether is added, the tube shaken well for a minute, and then allowed to stand for a minute. If the precipitate consists only of sulphur, the tube will contain two layers, the upper perfectly clear, and the lower very slightly turbid; but if traces of the metals of the second group are present, they will be observed as a thin coloured film at the junction of the two layers. C. A. M.

The Determination of Nitrogen in Nitrates, Nitro-compounds, and Nitroso-compounds. M. Krüger. (*Ber.* 1894, xxvii., 1633; through *Chem. Zeit.*)—0.2 to 0.3 gramme of the substance in which the nitrogen is to be determined is placed in a globular flask together with 20 c.c. of water (or an equal volume of alcohol if the

substance be soluble in that menstruum and difficultly soluble in water), and treated with 10 c.c. of an acid solution of stannous chloride and 1.5 gramme of spongy tin. The flask is then heated until the contents are colorless and the tin dissolved. (The alcohol, if present, is evaporated at this stage.) After cooling, 20 c.c. of strong sulphuric acid are added, and the mixture concentrated until fumes of sulphuric acid come off freely. When the liquid is again cool, potassium bichromate is added to oxidize amido-compounds, and complete resolution of the nitrogenous substances having thus been effected, the determination is finished in the usual manner. Substances which are volatile with steam even in acid solution and require the addition of alcohol, are thoroughly reduced by gentle warming on the water-bath until it is safe to evaporate off the alcohol in the manner mentioned above. A preferable plan, however, consists in carrying out the operation in a sealed tube immersed in a boiling water-bath.

B. B.

The Separation and Determination of Arsenic, Antimony and Tin. W. Hampe. (*Chem. Zeit.*, 1894, pp. 1900, 1901.)—The successful separation of these metals is always a matter of some difficulty, and depends in large measure on attention to details of manipulation rather than on the employment of any novel principle of separation.

Arsenic, antimony and tin are usually obtained in the course of an analysis as thio-salts, from which the sulphides can be recovered by precipitation with an acid. The precipitate of mixed sulphides, together with excess of sulphur, is dealt with in the following manner: The bulk of the precipitate is washed into a small beaker, and the residue on the filter is dissolved through by the least possible amount of the freshly-prepared ammonium sulphide into the same beaker. Whether the bulk of the precipitate dissolves or not is of no importance. The whole mass is treated with strong hydrochloric acid and oxidized with potassium chlorate; the chlorine is driven off, tartaric acid added, and the solution filtered. Excess of ammonia is added to the filtrate, which will remain perfectly clear, provided sufficient tartaric acid be present. More tartaric acid is needed when the mixture contains tin than when it contains only antimony. Arsenic is now precipitated by magnesia mixture from the solution kept at as low a bulk as possible, and the precipitated ammonio-magnesium arseniate is purified by solution in hydrochloric acid and precipitation with ammonia, the filtrate being added to the main filtrate, which is then acidulated with hydrochloric acid, and precipitated with sulphuretted hydrogen. From the mixed precipitate the sulphides of antimony and tin are separated in the following manner: The bulk of the precipitate is transferred to a beaker; the portion adhering to the filter-paper is dissolved through with freshly-prepared sodium sulphide used in as small quantity as possible, the washing being similarly restricted, so that the bulk of the solution in the beaker is but small. The total solution of sodium thio-salts is then treated with successive small quantities of sodium peroxide. It is to be noted that the sodium peroxide should be taken from the central parts of the tinplate canisters in which it is sold, as traces of tin from the metal of the canister might otherwise be introduced. As soon as the liquid has become colourless, and effervesces

on the addition of a further quantity of sodium peroxide, on account of the escape of oxygen, the oxidation of the thio-salts may be taken as complete, and the contents of the beaker then consists of caustic soda, sodium sulphate, sodium stannate and sodium antimoniate. The solution is boiled, allowed to cool, and treated with one-third of its volume of alcohol of specific gravity 0.833 to precipitate the last traces of sodium antimoniate. The beaker is roughly graduated as a guide to the addition of the proper quantity of alcohol. The solution is allowed to stand for 24 hours, and transferred to the filter with a mixture of one volume of alcohol and three of water, and washed according to Rose's prescription, namely, first with a mixture of one volume of water and one of alcohol of sp. gr. 0.833, and finally with one containing one volume of water and three of alcohol. All these washing liquids are made slightly alkaline by the addition of a drop of caustic soda. The sodium antimoniate on the filter is dissolved in hydrochloric acid containing tartaric acid, the solution precipitated with sulphuretted hydrogen, and the antimony sulphide filtered and washed. As much as possible is detached from the filter and gently ignited in a stream of carbon dioxide and weighed as the anhydrous tri-sulphide; the residue adhering to the filter is dissolved through by means of ammonium sulphide, the solution evaporated to dryness in a weighed porcelain dish, and the residue oxidized with nitric acid ignited and weighed as Sb_2O_4 . The filtrate from the sodium antimoniate is evaporated to drive off the alcohol, acidulated and precipitated with sulphuretted hydrogen. The tin sulphide is roasted to oxide and weighed. When a large quantity is present and the removal of alkali salts is consequently difficult, the precipitate is redissolved in ammonium sulphide and reprecipitated by sulphuric acid.

When a larger quantity of antimony than of arsenic and tin is present, as in hard lead, the antimony can be separated at the first stage of operations in the following way: The mixed sulphides are extracted with sodium sulphide and oxidized with sodium peroxide, the arsenic being converted into sodium arseniate and the sodium antimoniate being precipitated. On endeavouring, however, to precipitate the arsenic in the filtrate from the antimony by means of magnesia mixture, it is found that the large quantity of sodium salts in solution prevents the complete precipitation of the arsenic. It is necessary therefore to evaporate the filtrate from the sodium antimoniate to drive off the alcohol which it contains, then to precipitate the sulphides of tin and arsenic by acidulation with hydrochloric acid and by passing sulphuretted hydrogen, and finally to oxidize the sulphides with potassium chlorate and hydrochloric acid and separate them in the usual manner.

The method has been tried upon known mixtures and yields satisfactory results.

B. B.

Detection of Chlorine in presence of Iodine, and Quantitative Separation of these Halogens. P. N. Raikow. (*Chem. Zeit.*, 1894, xviii., 1661.)—The method is based on the formation of a precipitate when an aromatic hydrazine (*e.g.*, phenylhydrazine or tolylhydrazine) is mixed* with copper sulphate, sulphuric acid, and a soluble iodide, and on the absence of such a precipitate when a chloride is substituted for the iodide. On treating an aqueous solution of the sulphate of phenyl-

hydrazine with one of copper sulphate, a bulky gray-brown precipitate is formed. Should either of the solutions contain free sulphuric acid, however, no precipitate is produced. When hydriodic acid or a soluble iodide is present, a voluminous precipitate, reddish-gray in colour, is thrown down. The test is carried out as follows: A freshly-prepared solution of the sulphate of phenylhydrazine is acidulated with dilute sulphuric acid, and treated with a solution of copper sulphate in excess. To the clear liquid the aqueous solution containing the chloride and iodide is added, and the mixture is shaken. A precipitate collects on the surface of the liquid, which appears clear and blue in colour. The precipitate is collected and washed with cold water. It contains the whole of the iodine. The chlorine may be recognised in the filtrate by means of silver nitrate after the addition of a further quantity of sulphuric acid, which is necessary to hinder the reducing action of the phenylhydrazine on the silver salt. The iodine is determined by boiling the precipitate with dilute caustic potash, filtering from copper oxide, acidulating with sulphuric acid, and proceeding in the usual manner. The bromine compound obtained by the use of phenylhydrazine is intermediate in solubility between the chloride and iodine compounds, and thus the presence of bromine interferes with the success of the separation.

B. B.

On the Detection of Geranium-oil in Rose-oil. Robert Jedermann. (*Zeit. f. Analyt. Chem.*, 1895, p. 51.) As the result of an extended experience, the author has come to the conclusion that there is as yet no reliable test for the purity of rose-oil. Schiff's colour reaction and Hager's turbidity test are both unsatisfactory, and the freezing-point and specific gravity of the oil inconclusive. The specific gravity of rose-oil varies in different specimens according to the conditions of production and preparation. The pure stearoptene, which constitutes the scentless part of rose-oil, has a specific gravity of 0.813, while that of the oil free from stearoptene varies from 0.8730 to 0.8735. As the amount of this constituent in Balkan rose-oil varies from 7 to 30 per cent., and as the specific gravity of geranium-oil lies between 0.882 and 0.895, a rose-oil containing but little stearoptene, and consequently having a high specific gravity, is liable to be suspected of adulteration; on the other hand, the detection of very small quantities of geranium-oil is impossible.

The behaviour of the oil towards polarized light is also useless as a test, since rhodinol and geraniol are isomeric bodies, and behave similarly in this respect.

In consequence of the want of conclusive chemical and physical tests, the soap manufacturers and perfumers prefer to take the opinion of an expert who judges of the value of a sample by its odour.

C. A. M.